

## Environmental Protection Agency

## Pt. 60, App. A-4, Meth. 7B

pressure in the flask ( $P_f$ ) is the barometric pressure less the manometer reading.

8.2.3 Transfer the contents of the flask to a leak-free wash bottle. Rinse the flask three times with 10-ml portions of water, and add to the bottle. Mark the height of the liquid

level so that the container can be checked for leakage after transport. Label the container to identify clearly its contents. Seal the container for shipping.

### 9.0 Quality Control

Section	Quality control measure	Effect
10.1 .....	Spectrophotometer calibration .....	Ensures linearity of spectrophotometer response to standards.
11.4 .....	Audit sample analysis .....	Evaluates analytical technique and preparation of standards.

### 10.0 Calibration and Standardizations

Same as Method 7, Sections 10.2 through 10.5, with the addition of the following:

10.1 Determination of Spectrophotometer Standard Curve. Add 0 ml, 5 ml, 10 ml, 15 ml, and 20 ml of the  $\text{KNO}_3$  working standard solution (1 ml = 10  $\mu\text{g}$   $\text{NO}_2$ ) to a series of five 100-ml volumetric flasks. To each flask, add 5 ml of absorbing solution. Dilute to the mark with water. The resulting solutions contain 0.0, 50, 100, 150, and 200  $\mu\text{g}$   $\text{NO}_2$ , respectively. Measure the absorbance by ultraviolet spectrophotometry at 210 nm, using the blank as a zero reference. Prepare a standard curve plotting absorbance vs.  $\mu\text{g}$   $\text{NO}_2$ .

NOTE: If other than a 20-ml aliquot of sample is used for analysis, then the amount of absorbing solution in the blank and standards must be adjusted such that the same

amount of absorbing solution is in the blank and standards as is in the aliquot of sample used.

10.1.1 Calculate the spectrophotometer calibration factor as follows:

$$K_c = \frac{\sum_{i=1}^n M_i A_i}{\sum_{i=1}^n A_i^2} \quad \text{Eq. 7B-1}$$

Where:

$M_i$  = Mass of  $\text{NO}_2$  in standard  $i$ ,  $\mu\text{g}$ .

$A_i$  = Absorbance of  $\text{NO}_2$  standard  $i$ .

$n$  = Total number of calibration standards.

10.1.2 For the set of calibration standards specified here, Equation 7B-1 simplifies to the following:

$$K_c = 50 \frac{A_1 + 2A_2 + 3A_3 + 4A_4}{A_1^2 + A_2^2 + A_3^2 + A_4^2} \quad \text{Eq. 7B-2}$$

10.2 Spectrophotometer Calibration Quality Control. Multiply the absorbance value obtained for each standard by the  $K_c$  factor (reciprocal of the least squares slope) to determine the distance each calibration point lies from the theoretical calibration line. The difference between the calculated concentration values and the actual concentrations (*i.e.*, 50, 100, 150, and 200  $\mu\text{g}$   $\text{NO}_2$ ) should be less than 7 percent for all standards.

### 11.0 Analytical Procedures

11.1 Sample Loss Check. Note the level of the liquid in the container, and confirm whether any sample was lost during shipment. Note this on the analytical data sheet. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results.

11.2 Sample Preparation. Immediately prior to analysis, transfer the contents of

the shipping container to a 100-ml volumetric flask, and rinse the container twice with 5-ml portions of water. Add the rinse water to the flask, and dilute to mark with water.

11.3 Sample Analysis. Mix the contents of the flask thoroughly and pipette a 20 ml-aliquot of sample into a 100-ml volumetric flask. Dilute to the mark with water. Using the blank as zero reference, read the absorbance of the sample at 210 nm.

11.4 Audit Sample Analysis. Same as Method 7, Section 11.4, except that a set of audit samples must be analyzed with each set of compliance samples or once per analysis day, or once per week when averaging continuous samples.

### 12.0 Data Analysis and Calculations

Same as Method 7, Section 12.0, except replace Section 12.3 with the following:

12.1 Total  $\mu\text{g}$   $\text{NO}_2$  Per Sample.